TRANSLATION

PLANNING THE RECOVERY OF VOLTAILE SOLVENTS WITH BATCH-OPERATED ADSORBERS (SELECTED PARTS)

By

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FOREIGN TECHNOLOGY DIVISION

AIR FORCE SYSTEMS COMMAND

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MIXED SOLVENTS

[4], [8], [9], [20], [29], [30], [33], [36], [43], [45]

Mixed solvents are those which chemically are not separate compounds--gasolines, turpentines, solvent naphthas, and so forth--and also various mixtures of organic solvents of undetermined composition.

GASOLINES (Benzines)

Benzine (gasoline) solvents are obtained from the low boiling-point fractions of petroleum by direct distillation. In general they are mixtures of paraffin, naphthyl, and aromatic hydrocarbons beginning with gaseous propane and butane dissolved in heavier substances with five or more atoms of carbon. The composition of gasolines depends on their origin. If gasolines are obtained from various raw materials, they will not have exactly similar properties even distilled over the same temperature ranges.

The following are used as solvents: BR-1 ("Galosha"/lit. "overshoe"), BR-2, B-70, extractions, and white spirit.

Benzine BR-1 is produced from the low-sulphur Grozny and mountain paraffin petroleums; BR-2 from the sulphurous eastern paraffin petroleums. The technical specifications for BR-1 and BR-2 benzines are the same, but there has been established an additional standard for content of total and mercaptan sulphurs for BR-2, since it is prepared from sulphurous raw material. Total sulphur content is also fixed for B-70, extraction, and white spirit.

Benzine solvents (except B-70) must have a narrow boiling range. This is dictated by the technical peculiarities of their application. The comparatively high initial boiling temperature of the benzines BR-1, BR-2, and extraction (70-80°) reduces evaporation losses in production, and at the same time reduces the degree of toxic activity

and inflammability. The relatively low final boiling temperature (95-120°) facilitates their removal from working solutions. For the benzines BR-1 and BR-2, the totality of evaporation is characterized by testing for the formation of grease spots.

The benzines BR-1 and BR-2 are basically used in the rubber industry, while extraction is used in the feed industry.

White spirit, being heavier than benzine (boiling point in the range 165-200°) belongs to the high-boiling-point solvents. It is used for the preparation of paints and varnishes. Drying time and spreading quality (paint and varnish film) depend to a significant degree on the solvent's speed of evaporation; therefore, a volatility index was established for white spirit, based on the properties of xylene whose volatility was provisionally taken as the unit.

Compared with other benzine solvents, the benzine B-70 has the widest boiling range (40-180°). Its relatively low initial boiling temperature makes it much more toxic and inflammable. Benzine B-70 is used in the manufacture of leather substitutes, for chemical dry cleaning and other technical needs connected with solvent activity.

Reducing the toxicity of benzine solvents is of primary importance since they are widely used in industry and daily living. To this end their content of aromatic hydrocarbons is limited by technical specifications, nor is the presence of tetraethyl lead, the additive for ethylation of a number of motor fuels, permitted.

The specifics for using benzine solvents require that particular attention be paid to maintaining their quality. In storage, transportation, and production, benzines for other purposes are not allowed to be mixed with them. Nor are benzine solvents allowed to be exchanged for other types of benzines.

The technical specifications for benzine solvents and white spirit are given in appendixes 3 and 4.

Some characteristics for benzines BR-1, B-70, and white spirit are given in table two.

TABLE 2

CHARACTERISTICS OF BENZINES BR-1, B-70, AND WHITE SPIRIT

Properties	BR-1 ("Galosha")	B-70	White spirit
1. Specific gravity at 20°C in gr/cm^3	0.73	0.745	0.795
2. Limits of the boiling range in degrees C.	80-120	40-180	165-200
3. Flash point in °C.	-17	-34	33
4. Temperature of spontaneous combustion in degrees C	350	300	270
5. Concentrated explosive range of air-vapor mixtures in % of volume			
lower	1.1	0.79	1.4
higher	5.4	5.16	6.0

The freezing point of the benzines is below -100°C, the specific heat of liquid is about 0.5 kilocalories per kilogram per degree at room temperature, their solubility in 100 gr. of water, for example, is 0.007 gr. and the solubility of water in the benzines is less than 0.05% by weight.

The molecular weight of benzine, its specific heat in the liquid and gaseous states, its heat of vaporization, and its heat conductivity may be approximately defined by the following empirical formulae [28], [40]:

average molecular weight according to Voinov

$$M = 59 + 0.3t_{BOIL} + 0.001t^2_{BOIL};$$

true specific heat of the liquid product according to Kragoya

$$C_t = \frac{0.403}{\sqrt{d}} (1 + 0.002t_{LIQ}) \quad \text{Cal/kg·degree}$$

real specific heat of the vapors at atmospheric pressure, according to Balk and Kay

$$C_p = \frac{4 - d}{6450} (1.8t_v + 720) \quad \text{Cal/kg·degree;}$$

closed vaporization heat at atmospheric pressure, according to Kragoya

$$r = \frac{1}{d} (60 - 0.09t_{BOIL}) \text{ Cal/kg;}$$

coefficient of heat conductivity of the liquid product, according to Kragoya

$$\lambda_t = \frac{0.1008}{d} (1 + 0.00054t_{LIQ}) \text{ Cal/m·br.degree.}$$

In the formulae

d - specific weight of the liquid product at 15°C in gr/cm^3;

t_{LIQ} - temperature of the liquid product in degrees C;

t_{BOIL} - average boiling temperature in degrees C;

t_v - temperature of the vapors in degrees C.

Since benzines are not chemically separate compounds, their vapor pressures vary not only according to their temperatures but also by the composition of their liquid and gaseous phases and by the ratio of the volume in which evaporation occurs to that of the liquid. The larger this ratio, the lower the vapor pressure. The relationships of separate hydrocarbons in the gaseous and liquid phases differ with respect to the pressure and vaporization temperature. At lower temperatures the vapors are richer in light components than at higher temperatures.

Unlike substances of a single chemical composition, the distillation temperature of benzines does not remain constant but has a gradual

unbroken increase as a result of the ever-increasing weight of the distillation fractions throughout. During this passage, the liquid temperature is higher than that of the vapor.

According to the conditions indicated, in determining the vapor pressure of benzine it is necessary to work from experimental data. The use of empirical formulae and analogies provides only rough guidelines.

TURPENTINES

The products known under the name of turpentines are obtained by various methods from trees of the coniferous variety: resinous or sulphuric turpentine (turpentine oil)--from the resinous parts (soft resins, pitch) of the growing trees; stump turpentine--from the roots, stumps, and branches; wood turpentine--from the waste products of the wood; and cellulose turpentine--from the manufacture of cellulose by the sulphate and sulfite methods. The quality and composition of turpentines differ according to their origins and methods of production. Basically they consist of hydrocarbons of the terpene order. The resinous turpentines are of the highest quality.

Russian resinous turpentine contains mainly detro-pinene with two double bonds, carene and high fractions (sesquiterpenes, oxidation products, etc.). It should have a characteristically pleasant smell, be colorless or have a slight yellowish tint, and also satisfy the following technical specifications.

TECHNICAL SPECIFICATIONS FOR TURPENTINE
(TURPENTINE OIL)

External appearance.	transparent, volatile liquid with characteristic odor, no sediment or water.
Specific gravity d_4^{20} in gr/cm^3	within 0.855-0.863
The color of a column of turpentine 220 mm. high should be no darker than that of a standard solution of potassium bichromate of a height of (in. mm.).	not more than 90
Index of refraction	within 1.467 - 1.472
Initial boiling point in degrees C. . .	within 153-160
Volume of distillate up to temperature 170°C in %	not less than 92
Remainder after evaporation in % by weight.	not more than 0.5
Acid number in mg. KOH per 1 gr of oil.	not more than 0.7
Vapor pressure at 20°C.	~4.5 mm. of mercury
Evaporation speed compared with xylene.	not more than 4
Specific heat at 20°C	~0.5 Cal per kg per degree
Heat of evaporation	~68 kilocalories (Cal)/kg
Flash point	~34°C
Spontaneous combustion point.	~300°C
Lower limit of concentration for explosion of the vapors in air. . . .	0.8% by volume

Turpentine easily absorbs oxygen from the air forming peroxide compounds. Heating and the action of light facilitate this. Ultimately the peroxides separate, liberating oxygen as a result of which turpentine acts as an oxidizer.

Turpentine is used in the paint and varnish industry. It is one of the most important solvents in the production of oil varnishes and paints since it speeds up their drying.

SOLVENT NAPHTHAS

Solvent naphtha is obtained from coal tars and in particular from the wastes after cleansing and rectification of raw benzene. It is basically a mixture of hydrocarbons of the aromatic variety. It is made up of xylene (dimethylbenzene) and cumene (i-propyl benzene) (up to 95%), toluene and naphthalene oil (about 5%) and small quantities of conversion and naphtenic hydrocarbons. Toluene is found in the lighter product and naphthalene oil in the heavier.

The characteristics of two types of solvent naphthas are given in table 3. The solvent naphtha of the first type has a flash point of 34°, a spontaneous combustion temperature of 520°, an explosive concentration limit in air in Volume percentage on the low end, of 1.3% and on the high - of 8%. It is not soluble in water.

TABLE 3

CHARACTERISTICS OF SOLVENT NAPHTHAS

Properties	Type	
	1	2
1. Specific gravity at 15°C in gr/cm^3	0.86-0.88	0.87-0.91
2. Color and tranparency	Colorless or light yellow, transparent	Colorless or light yellow, darkening or reddening is accepted
3. Boiling point at 760 mm. of mercury	In the range 120-160°C, not less than 90% distills	Within the range 135-180°C, not less than 90% distills
4. Degree of purity (acid coloration on the Kremer-Spilker scale)	not more than 5.0	Not standardized

Besides solvent naphthas from coal, there are, for use as solvents (paints and varnishes), petroleum solvents obtained in the process of pyrolysis of petroleum products in rectifying and cleansing light oils.

The petroleum solvent is a mixture of aromatic hydrocarbons of the benzene variety.

TECHNICAL SPECIFICATIONS FOR PETROLEUM SOLVENT

External aspect.	colorless, transparent liquid, light yellow tint permitted
Specific gravity in d_4^{20} in gr/cm^3	0.875 ± 0.03
Initial distillation at (°C)	not lower than 135
End of distillation of 95% of liquid by volume at temperature in degrees C.	not higher than 200
Reaction to aqueous infusion	neutral
Volatility compared to xylene.	not more than 2
Oily spot test	on filter paper, it should leave no oily spot
Sulfonation substances in % by volume. .	not less than 85
Sulphur compounds (in conversion to sulphur) in %.	not more than 0.10

Note: Solvent naphthas should not contain water.

ACETONE ALCOHOL

This solvent is obtained as a side product of the dry distillation of wood in the production of acetone and methyl alcohol. It is a colorless, transparent, freely fluid liquid consisting of a mixture of acetone, methyl alcohol and ethers of acetic acid and its homologues. A light yellow color is permissible. When acetone alcohol is mixed with four times its volume of distilled water, a slight cloudiness is permitted. It should have a neutral or slightly acid reaction which, in translation to formic acid, should not exceed 0.015%.

The broad composition of acetone alcohol: acetone 35-60%, methyl alcohol 20-40%, methyl acetates and others 20-30%.

For the product containing not less than 55% acetone, the specific gravity at 20° should not be more than 0.83 gr/cm^3; up to 70°, not less

than 95% by volume should distill and a content of dry residue greater than 0.01% is not permissible.

Acetone alcohol is used in the production of lacquers for dissolving resins and nitrocellulose (in mixtures with solvents of high boiling points). Since methanol, which enters into the composition of acetone alcohol, is moderately poisonous, all precautionary measures are necessary when working with it.

MIXTURES OF SOLVENTS OF UNDETERMINED COMPOSITION

Several branches of industry put out under various trade marks and technical names solvents and dilutants in the form of mixtures containing two or more components. The composition of such mixtures is often unknown. For a large number of these mixed solvents and dilutants, the literature gives only specific gravity, boiling point, and solubility in water. Other characteristics are only rarely given.

APPENDIX 2

SOME TECHNICAL AND COMMERCIAL SYNONYMS FOR THE CHEMICAL NAMES OF SOLVENTS

Synonyms	Chemical names
AVANTIN	Isorpropyl alcohol
ADRONOL	Cyclohexyl alcohol
AKTILOL	Ethyl ether lactate
ALKILGLIKOL' (Alkylglycol)	Simple monoderivative ethers of ethylene glycol (Example: monomethyl glycol)
ANZOL M	Anhydrous ethyl alcohol
ANOL	Cyclohexyl alcohol
ASORDIN	Carbon tetrachloride
ATSETAL' (Acetyl)	Diethyl acetyl
BUTAKTOL	Butyl lactate
BUTIRON	Dipropyl ketone
BUTOKSIL	Methoxy acetyl acetate
BUTOL	Butyl butyrate
VESTROZOL	Ethylene trichloride
VESTRON	Ethane tetrachloride
GEKSALIN (Hexaline)	Cyclohexyl alcohol
GEKSANON (Hexanone)	Cyclohexanone
GEKSON (Hexone)	Methyl isobutyl ketone
GEPTALIN	Methyl cyclohexyl alcohol
GIDRALIN (Hydraline)	Raw cyclohexyl alcohol
GLIKOL' (Glycol)	Ethylene glycol
DEKALINE	Decahydronaphthalene
DIAL	Diacetone alcohol
DIATOL	Carbon diethyl ether

APPENDIX 2--CONTINUED

Synonyms	Chemical names
DIELIN (Dieline)	Ethylene dichloride
DIONAL	Mixtures containing acetyl alkyl ethers
DISSOL'VAN SA/CA	Diethyl acetyl
KARBITOL (Carbitol)	Monoethyl ether of diethylene glycol
KARBITOLY (Carbitols)	A general name for simple ethers of diethylene glycol (For example: monomethyl carbitol, diethyl carbitol)
KATARIN	Carbon tetrachloride
KUMOL	Isopropyl benzene
MASLO NIOBEI (Oil of Niobeya)	Methyl benzoate
M. E. K.	Methyl ethyl ketone
METILADRONOL (Methyladronol)	Methyl cyclohexyl alcohol
METILAL' (Methylal)	Dimethyl acetyl
METILANOL (Methylanol)	Methyl cyclohexyl alcohol
METILANON (Methylanon)	Methyl cyclohexanone
METILGEKSALIN (Methylhexaline)	Methyl cyclohexyl alcohol
METLANOL	The same
NORMANOL	Ethyl ether lactate
OKSIBUTIRAT (Oxibutyrate)	Oxy-iso-ethyl butyrate
PENTAZOL	Synthetic amyl alcohol
PENTALIN	Ethane pentachloride
PERSPIRT	Isopropyl alcohol
PERKHLORETILEN (Perchlorethylene)	Ethylene tetrachloride
PETROGOL	Isopropyl alcohol

APPENDIX 2--CONTINUED

Synonyms	Chemical names
PIRANTON A	Diacetyl alcohol
PLASTOLIN I	Benzyl acetate ether
PLASTOFORM I	Benzyl alcohol (phenyl carbinol)
PRESTON (Prestone)	Diethylene glycol
RASTVORITEL' BP (solvent)	Butyl propionate
RASTVORITEL' GA (solvent)	Diacetone alcohol
RASTVORITEL' GC (solvent)	Glycoacetyl monoderivative ether
RASTVORITEL' GD	Complete glycoacetyl ether
RASTVORITEL' GO	The same
RASTVORITEL' THD	Ethylene chloride
SEKSTAT	Acetyl methyl cyclohexyl ether
SEKSTOL	Cyclohexyl alcohol
SEKSTON	Cyclohexanone
SEKSTON B	Methyl cyclohexanone
SERNYI EFIR (Sulphuric ether)	Diethyl ether
SINTOL	Synthetic methyl alcohol
SOLAKTOL	Ethyl ether lactate
SOLASTIN	Methylene dichloride
SOL'VULEZ	Monoethyl ether of ethylene glycol
SPEKTROL	Carbon tetrachloride
SPIRT KOLUMBA (Columbus' alcohol)	Methyl alcohol
TAMAZOL	Butyl alcohol
TAMAZOL J	Butyl acetate
TETRA	Carbon tetrachloride
TETRAKOL	The same

APPENDIX 2--CONTINUED

Synonyms	Chemical names
TETRALIN	Tetrahydronaphthalene
TETRAFORM	Carbon tetrachloride
TRIELIN	Ethylene trichloride
FENIKSIN (Phoenixine)	Carbon tetrachloride
FORMAZOL	Ethyl formate
TSELLOZOL'V	Monoethylene ether of ethylene glycol
TSELLOZOL'VY	General name of the simple monoderivatives of the ethers of ethylene glycol (For example: monoisopropylene glycol)
ESTIZOL	Ethyl ether lactate
ETELIN (Ethylene)	Ethylene tetrachloride
EUSOL'VAN	Ethyl ether lactate
EFIR P (Ether P)	Ethyl proprionate

APPENDIX 3

TECHNICAL SPECIFICATIONS FOR BENZINES BR-1, BR-2, AND EXTRACTION

45

Properties	BR-1 ("Galosha")	BR-2	Extraction
1. Density at 20° compared to water at 4°	0.730	0.730	0.725
2. Fractional composition			
a) Begin boiling, in degrees Centigrade, not less than	80	80	70
b) Up to 95° distills in % not less than	--	--	98
c) Up to 110° distills in % not less than	93	93	--
d) Up to 120° distills in % not less than	98	98	--
e) Remainder in the flask after distillation in % not more than	1.5	1.5	1.0
3. Iodine number in gr. of iodine per 100 gr. of benzine not more than	0.10	0.10	--
4. Aromatic hydrocarbons in % not more than	3.0	3.0	4.0
5. Sulphur in %, not more than	--	0.025	0.025
6. Mercaptan sulphur	--	--	--
7. Water soluble acids and alkalis	--	--	--
8. Foreign matter and water	--	--	--
9. Test for formation of a grease spot	Passes	Passes	--
10. Content of tetraethyl lead	--	--	--

APPENDIX 4

TECHNICAL SPECIFICATIONS OF BENZINE B-70 AND WHITE SPIRIT

45

Properties	B-70	White spirit
1. Density at 20° with respect to that of water at 4°C, not more than	--	0.795
2. Fractional composition a) Begin boiling in °C, not lower than	40	Not higher than 165
b) 10% distills at temp. in °C not higher than	88	--
c) 50% distills at temp. in °C not higher than	105	--
d) 90% distills at temp. in °C not higher than	145	--
e) 97.5% distills at temp. in °C not higher than	180	--
f) distills up to temperature of 200° in %, not less than	--	98.0
g) residue and losses in % not more than	2.5	2.0
h) residue and flask in %, not more than	1.5	--
3. Volatility speed compared to xylene	--	3-4.5
4. Iodine number in gr. of iodine per 100 gr. of benzine, not more than	2.0	--
5. Aromatic hydrocarbons in %, not more than	--	16
6. Gums in 100 ml. of benzine in mg. not more than	2.0	--
7. Acidity in mg. KOH per 100 ml. of benzine, not more than	1.0	--
8. Sulphurs in %, not more than	0.03	0.025
9. Flash point (determined in a closed crucible) in °C, not lower than	--	33
10. Water soluble acids and alkalis	--	--
11. Foreign matter and water	--	--

APPENDIX 5

TECHNICAL SPECIFICATIONS FOR RECOVERY CARBON AR

Activated recovery carbon AR comes in hard, cylindrical granules prepared by pressing a bulk mass made of anthracite dust and tar and subsequent thermal processing in special commercial ovens.

Recovery carbon is destined for use in recovery technique.

TECHNICAL REQUIREMENTS

1. The activity on benzene of the dry carbon AR at a speed of vapor-air mixture of 0.5 liters per cm^2 per minute and at benzene concentrations of 30 ± 3.0 mg. per liter must be:

a) dynamic activity (calculated by the overweight of the "control" dynamic tube) not less than 115 gr. per liter;

b) static activity on benzene not less than 135 gr./liter.

2. Granulation. The prepared product must have granule dimensions in the range from 2.75 to 5.5 mm, which is determined by screening, and at which in the industrial mix there must be:

fractions larger than 5.5 mm . . . . . not more than 1%

fractions from 1.0 to 2.75 mm. . . . . not more than 15%

fine pieces less than 1 mm, and dust . not more than 1%

3. Mechanical strength against pulverization--not below 80%.

4. Weight of 1 liter of carbon--not less than 600 gr.

5. Moisture content--not more than 5%.

Note: At moisture content of the carbon up to 10%, the carbon is not rejected, but a commercial recalculation to moisture content of 5% is made.

PACKAGING AND MARKING

The finished AR is packed in iron drums or wooden boxes, thus assuring preservation of the product during storage and transportation.

Note: Packing recovery carbon in drums or boxes previously used is permissible.

On every drum or box is glued a paper label with the following marking:

a) Brand of carbon

b) Number of the consignment

c) Weight, brutto (gross)

d) Tare weight

e) Date of preparation

f) Name of the manufacturer

REGULATIONS FOR RECEPTION AND SAMPLING

Average samples are taken from a consignment of recovery carbon presented for delivery.

The quantity of material to constitute an average sample must be no less than 10% of the quantity of packages delivered, but not less than three samples.

The weight of each sample is 300-350 grams. The sample may be obtained with a shovel when the carbon is being poured from one package to another, or by a metal probe. The samples taken are mixed and from the mix about one kilogram of carbon is taken for analysis of activity, granulation, strength, and for determining the weight of a liter and the moisture content.

If on testing the properties of the carbon unsatisfactory results are obtained on one or more points, a second test is made with double the quantity of sampled material. If unsatisfactory results are obtained from the second analysis, the consignment is rejected.

TESTING METHODS

1. Testing the dynamic activity of recovery carbon with benzene.

Prior to testing it for dynamic activity, activated recovery carbon is dryed in a drying cupboard for three hours at a temperature of 110-120°. The test is made in a glass dynamic tube with a

cross-section of 5 ± 0.5 cm^2. The height of the carbon layer in the tube is 10 cm.

The dynamic tube is filled with activated carbon in three or four separate batches. After each batch, the carbon is tamped down to a constant volume with the aid of a little rubber hammer.

When it has been filled, the tube with the carbon is scavenged with clean dry air for 3 to 5 minutes after which it is weighed on a techni-scale with an accuracy to 0.01 grams and placed in the thermostat together with the flask, weighed beforehand. The temperature in the thermostat is kept at 15°.

Previously dryed air is, from time to time, admitted into the mixer while the remainder passes through the flask with the benzene whence it carries with it benzene vapor, then also enters into the mixer. From the mixer, the vapor-air mix passes into the dynamic tube charged with the carbon to be tested. The speed of the air-vapor mixture is 5 $l/cm^2 \cdot$minute, its concentration 30 ± 3.0 mg/liter.

The passage of the benzene is determined by the overweight in a "control" dynamic tube filled with AG carbon to a depth of 10mm. After being charged, the "control" is scavenged with dry air, weighed, and joined to the dynamic tube with the carbon to be tested.

The "control" is weighed the first time 60 minutes after the start of the test; later on it is weighed every 10 minutes until an additional weight of 0.01-0.02 gr. is obtained, and then every five minutes. The test is terminated when the overweight in the "control" attains 0.05-0.1 gr. relative to its weight before starting. After this the tested tube is removed and weighed.

The dynamic activity is calculated by the formula:

$$A = \frac{a \cdot b}{M},$$

in which A is the dynamic activity in gr/lit;

a - the weight of the tube with the tested carbon in gm;

M - the weight of the carbon taken for testing, in gm.;

b - the gravimetric specific gravity of the tested carbon in gm/lit.

A weight difference of ~~nor~~ not more than 6 grams is permitted between the two parallelly tested tubes.

2. Determination of the static activity of the recovery carbon by benzene.

After determination of the dynamic activity, the tube with the tested carbon is placed in the apparatus and the air-vapor mixture is passed through it until the carbon is completely saturated. This latter is determined by bringing the tube with the carbon to a constant weight.

The size of the static activity is calculated in gm/lit by the formula indicated above.

3. The granule size is determined by screening.

4. The mechanical strength is determined by grinding in a ball mill.

CONDITIONS OF TRANSPORT AND STORAGE

For protection of the packages and prevention of the possibility of grinding the carbon during transport it is necessary to:

a) avoid long voyages by land transport;

b) avoid throwing the drums and boxes in transferral and stacking;

c) keep the recovery carbon in a closed, dry location with a floor;

d) keep the drums or boxes of carbon sealed in warehouses by consignment.

Note: The technical specifications for recovery coal AR have been in effect since 1958. Since recovery coal is an article of many years duration of service (5-7 years on the average), the technical specifications provided can serve as a useful way of checking the value of carbon on hand.

Since 1958 active recovery carbon AR-3 has been made for recovery installations, according to GOST 8703-58.

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